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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of: TIMOTHY WARNER	Group Art Unit: 1742 Examiner: J.A. Morillo
Serial No.: 10/066,788	
Filed: February 6, 2002	

For: MANUFACTURING PROCESS FOR A HIGH STRENGTH WORK HARDENED PRODUCT MADE OF AlZnMgCu ALLOY

DECLARATION UNDER 37 CFR 1.131

Honorable Commissioner for Patents
PO Box 1450
Alexandria, VA 22313-1450

Sir:

I, Timothy Warner, do hereby declare as follows:

I am the named inventor of the above-identified patent application.

I am familiar with published US Application No. 2002/0121319 A1 to Chakrabarti et al, published September 5, 2002, and based on a US patent application filed on October 4, 2001. This application is a continuation-in-part of a US patent application filed on January 31, 2001.

The present application was filed in US Patent and Trademark Office on February 6, 2002, and claims the priority of French application 01/01617 filed February 6, 2001. While the French priority date is subsequent to the Chakarabarti et al parent filing date, the invention disclosed and claimed in the present patent application was completed prior to the filing date of the Chakrabarti et al parent application (January 31, 2001), and subsequent to January 1, 1996, in a WTO

country.

In support of the asserted date of invention, submitted herewith are three reports on which I am named as a co-author, labeled Exhibit A, Exhibit B and Exhibit C. All three reports are verified by me to be true and accurate translations of original French language reports and are redacted as to dates and non-relevant subject matter.

1) In Exhibit A: a) The introduction outlines the problem solved by the patent, optimizing compressive yield strength in AA 7x49 alloys. b) The experiments described on pages 18-20 are very similar to those of example 1 and 2 of the patent application. They show that the optimum ageing treatment for tensile yield stress is not the same as for compressive yield stress. This effect is set forth on pages 26-28 of the report as the difference R(tens)0.2(L) - R(comp)0.2(L); the various parameters which have an influence on this difference are identified and explained as being related to the so-called remanent Bauschinger effect. c) The conclusions on page 32 of the report clearly point towards the solution given in the patent application, the main influence on the effect is the equivalent ageing time, although the aging times reported are less than those of the present claims; the maximum aging time reported was 96 hours at 120°C (page 21, paragraph 3.4).

d) Table 2 on page 13 gives information on the permanent set of the alloys. 2) In Exhibit B: a) The starting point of this report is the conclusion of the previous report (Exhibit A), the optimum compressive yield stress should be obtained using an ageing treatment intermediate between existing T651 and T7951 practices (see patent application, page 5, lines 13-18). This leads to an improvement of +25 MPa with respect to T7951 (report page 4; patent application, page 12,

lines 3-12).

b) Page 3 of this report gives the composition of the plates used for the study, which falls within the composition ranges of the present claims, and describes the aging treatments. The study results based on the aging treatments is given on page 8.

c) Page 4 discusses aging times of 24 to 36 hours at 135°C, which corresponds to 108 to 135 hours at 120°C, within the ranges of the claimed invention.

3) In Exhibit C: a) Figure 1.1 of the report includes all data of Figure 4 of the patent application, including aging times in excess of 100 hours at 120°C.

b) Table 2.1 of the report identifies the alloys (plate nos. 708695 and 716937) of table 4 of the patent application.

c) Figure 3.1 of the report, also section 4 "Discussion," contains the two- and three step ageing practices of claims 11 and 12.

d) The alloy compositions tested and ageing treatments are set forth in Section 2.1 Materials on page 4 of the report.

e) Page 10 of this report gives a detailed description of the steps used in treating the alloy, including the value of the permanent set.

The parallelogram recited in claim 7 of the present application was not discussed in the reports, but was based on an analysis of data found in the reports.

The date on which each of the reports of Exhibits A, B and C was issued was prior to January 31, 2001 and subsequent to January 1, 1996. These reports represent work done in a WTO country.

Based on the data in the reports discussed above, I believe that I was substantially in possession of the claimed invention prior to the filing date of the Chakrabarti et al

reference, January 31, 2001.

I further declare that all statements made by me herein are true and all statements made on information and belief are believed to be true, and that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and may jeopardize the validity of the application or any patent issued thereon.

June 30th 2005
Date

Timothy Warner

AUG 0 5 2005
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PECHINEY

CRV

Archiving ID	Microfilming
Yes [redacted]	Yes [redacted]

EXHIBIT A

Your Ref.:

Our ref.: CRV/ALT/TJW

Parameters governing the compression yield stress of alloys for wing upper surfaces

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Author:

T. WARNER CRV/ALT

J. DOMEYNE CRV/ALT

To: [redacted]

For info: [redacted]

Project: 4537

Requested by: RHU

Abstract

The purpose of this note is to identify and hierarchise the main parameters governing $R_{0.2}^{comp}(L)$ of a plate for wing upper surfaces. The specific features of the definition of measurement of $R_{0.2}^{comp}(L)$ are described in §2. Experimental results for parameters that could make a different contribution to $R_{0.2}^{comp}(L)$ and to $R_{0.2}^{tens}(L)$ are then considered (§3 and §4). It is found that the value of $R_{0.2}^{tens}(L) - R_{0.2}^{comp}(L)$ will reduce as:

[redacted]

- the equivalent ~~annealing~~ ageing time increases (see §3.3);
- dissolution of the alloy improves (see §3.4);
- the ~~tension ratio~~ permanent set reduces (temper T651) (see §3.2).

There is also an influence of the granular structure (see §3.1), but the important parameter is not only the local recrystallisation ratio. [redacted]

Therefore, to optimise $R_{0.2}^{comp}(L)$, [redacted] and the composition must be suitable for the solution heat treatment. Note that there is an inherent asymmetry in the use of a conventional yield stress such that the difference $R_{0.2}^{tens}(L) - R_{0.2}^{comp}(L)$ is negative ($\approx$ - 13 MPa for 7449) if there is no difference between the rational tension and compression curves.

Rationalisation of these observations is proposed in §5: the asymmetry of the yield stresses could be the result of a remanent Bauschinger effect due to the development of micro-stresses during stress relaxation by tension. This remanent Bauschinger effect would reduce during ~~annealing~~ ageing due to relaxation of these micro-stresses. Furthermore, an analysis of the mechanisms involved suggests that the ~~annealing~~ ageing peak for $R_{0.2}^{comp}(L)$ probably corresponds to over~~annealing~~ ageing with respect to $R_{0.2}^{tens}(L)$.

Recommendations

1 - For future productions of 7449-01:

- roll the plates at the highest possible temperature to minimise the recrystallisation ratio;

[redacted]

2 - Perform an ~~annealing~~ ageing procedure for $R_{0.2}^{comp}$ (L) since the ~~annealing~~ ageing peak for this parameter probably corresponds to over~~annealing~~ ageing with respect to $R_{0.2}^{tens}$ (L). This procedure should be characterised on at least two plates if possible, namely plates with high and low values of $\Delta R_{0.2}$ in the T651 temper, to quantify the gain in $R_{0.2}^{comp}$ (L).

3. Complete this work by tests to help understand and explore optimised solutions for the stress relaxation of wing plates.

1. Introduction

The compression yield stress in the longitudinal direction ($R_{0.2}^{comp}(L)$) is the controlling criterion for some commercial aircraft parts, particularly the skin and upper surface stiffeners. At the present time, production tests on wing panels do not include a measurement of $R_{0.2}^{comp}(L)$ since the value $R_{0.2}$ in tension was considered as being sufficient for testing and design. However, in the future, our main customer for these wing skins [redacted] will design the parts concerned using the compression stress-strain curve[1], and consequently would become more interested in the compression yield stress *rather than the tension yield stress*. At the present time, $R_{0.2}^{comp}(L)$ is lower than $R_{0.2}^{tens}(L)$ for our 7449 plates, particularly in the T651 temper (difference of 20 to 40 MPa) and to a less extent in over~~annealed~~ aged tempers (difference equal to 0 to 15 MPa). Note also that extruded products have the opposite behaviour ($R_{0.2}^{comp}(L) > R_{0.2}^{tens}(L)$), like 7055 - T7751 according to Alcoa data[2]. Furthermore, anisotropy of the yield stress is greater for 7449 - T651, for example than for 7150 - T651. Therefore, it is clear that anisotropy of the yield stress can be modified and that it is useful to do this depending on the product specification.

[1] Telephone discussion with [redacted]
[2] Alcoa technical fact sheet on 7055-T7751(1)

The purpose of this note is to identify and hierarchise the main parameters governing $R_{0.2}^{comp}(L)$ for a rolled product. The specific features of the definition and measurement of $R_{0.2}^{comp}(L)$ are described in §2. Experimental results concerning parameters that could make a different contribution to $R_{0.2}^{comp}(L)$ and to $R_{0.2}^{tens}(L)$ are considered in §3; granular structure, ~~tension ratio~~ permanent set, time between quenching and tension, ~~annealing~~ ageing, composition of the alloy. The information obtained from these results is used in the analysis of compression yield stresses that were obtained on industrial plates (see §4). Finally, an attempt to rationalise observations is given in §5.

2. Measurement of the compression yield stress

Procedures for measuring the compression yield stress used at the CRV and Issoire are consistent with external standards, and particularly with ASTM standard E9. The slenderness ratio of the test piece satisfies the geometric conditions necessary to avoid buckling and more generally has the geometry recommended for the measurement of $R_{0.2}$.

The yield stress is determined from the stress-strain curve in exactly the same way for the tension and compression curves. However, it is interesting to note two significant differences between $R_{0.2}^{tens}(L)$ and $R_{0.2}^{comp}(L)$ related to the elastic - plastic behaviour of

the material. Firstly, an increase in the cross section of the test piece is observed associated with elastic deformation when the material is in compression, unlike the situation for tension. This can be represented as follows:

$$\frac{\Delta S}{S} = \left(1 + \nu \frac{\sigma_{11}}{E} + 0.5\varepsilon_p\right)^2 - 1 \qquad \text{Eq. 1}$$

(where S is the area of the sample, ν is Poisson's ratio for Aluminium ($\approx$ 0.34), E is the modulus of elasticity (= 72 GPa)), which for a material with a yield stress of about 600 MPa corresponds to an increase in area of:

$$\frac{\Delta S}{S} = 0.0076 \qquad \text{Eq. 2}$$

By symmetry, the reduction of the section in tension at 0.2% would be of equal magnitude. Therefore a comparison of the conventional yield stresses in tension and in compression would include **an intrinsic bias of about 1.5% (namely about 9 MPa for a material with $R_{0.2}$ = 600 MPa) with higher values for compression,** if "real" yield stresses are identical.

There is also an intrinsic bias in the use of the nominal deformation compared with the true deformation. The difference between total true deformations at the point at which the yield stress is measured (conventional plastic deformation = 0.2%) is:

$$\Delta\varepsilon_{true} = \ln\left(1 + \frac{R_{0.2}}{E} + 0.2\%\right) + \ln\left(1 - \frac{R_{0.2}}{E} - 0.2\%\right)$$

$$\approx \left(\frac{R_{0.2}}{E} + 0.2\%\right)^2 \qquad \text{Eq. 3}$$

This difference is about 1×10^{-4} for an alloy with a yield stress of about 600 MPa.

For a slope ($d\sigma/d\varepsilon$) with 0.2% plastic deformation equal to about 20-30 GPa, this effect results in **an additional asymmetry of about 2 - 3 MPa.**

Moreover, a difference is observed between the shapes of the tension and compression curves in the longitudinal direction. This is illustrated in Figure 1 for different products; 38 mm thick plates made of 7449 - T651 and 7449 - T7951, and wing sections made of 7449 - T6511 and 79511. Significantly greater micro-plasticity will be observed for compression than for tension, for T6 in compression than for T79 in compression, and for the plate compared with the section. **Therefore the use of a yield stress at 0.1% or even 0.05% for a plate in the T651 temper would have a very bad effect on the tension - compression asymmetry.**

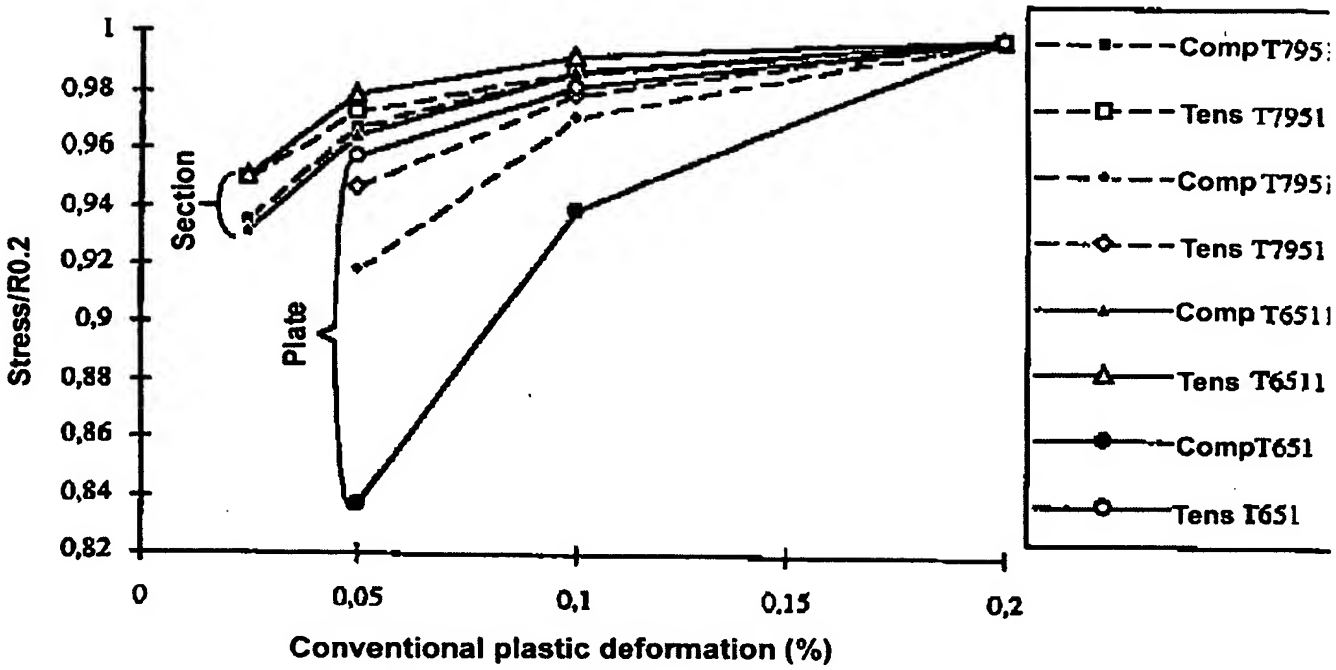


Figure 1. Illustration of the difference between curve shapes in tension and in compression in the longitudinal direction in temper Tx51 (I)

3. The predominant parameters

3.1. The granular structure

A first indication of the effect of the granular structure on the asymmetry of $R_{0.2}(L)$ in compression and in tension is obtained by comparing this asymmetry for a section and for a plate (see table 1). It is observed that:

- $R_{0.2}^{tens}(L)$ is systematically higher than $R_{0.2}^{comp}(L)$ for plates;

- on the other hand, $R_{0.2}^{tens}(L)$ is systematically lower than $R_{0.2}^{comp}(L)$ for sections. The average difference $R_{0.2}^{comp}(L)$ - $R_{0.2}^{tens}(L)$ is 13 MPa, which corresponds approximately to the asymmetry expected due to the use of the conventional curve in the assumption for identical rational curves for tension and compression (12 MPa, see equations 2 and 3).

- $R_{0.2}^{tens}(L)$ is systematically lower than $R_{0.2}^{comp}(L)$ for plates and sections. In this case, the difference is significantly more than 12 MPa.

- asymmetries vary with annealing. This point will be dealt with in §3.3.

Therefore, it appears that asymmetry between yield stresses in compression and tension varies as a function of the product. Considering the similarity between finishing phases of these two products, it would seem logical that the asymmetry is a result of the granular structure of products.

Table 1. Yield stresses in tension and compression, L and TL directions, for sections and for plates made of 7449

Product	$R^{tract}_{0,2}$(L) (MPa)	$R^{comp}_{0,2}$(L) (MPa)	$\Delta R_{0,2}$(L) (MPa)	$R^{tract}_{0,2}$(TL) (MPa)	$R^{comp}_{0,2}$(TL) (MPa)	$\Delta R_{0,2}$(TL) (MPa)
Results on sections (wing stringers)						
7449-T6511 section 67149*	684	693	-9	597	653	-56
7449-T6511 section 67154*	694	705	-11	589	646	-57
7449-T79511 section 67149*	623	644	-21	579	614	-35
7449-T79511 section 67154*	637	648	-11	592	617	-25
Results on 38mm plate (samples taken at mid-thickness)						
7449-T651 plate 665101	631	614	17	620	653	-33
7449-T651 plate 684978	627	587	40	618	not measured	not measured
7449-T7951 plate 665101	602	603	-1	604	not measured	not measured
7449-T7951 plate 684978	594	587	7	591	not measured	not measured

* values measured in this table are the average of two samples (taken at the beginning and end of extrusion), after two types of quenching (F223 or F227). The shape of the 67149 section corresponds to IS2229 (intrados), and 67155 to IS 2217 (wing upper surfaces).

In order to clarify this point, we carried out tests on two 70 mm thick 7010 plates derived from the same casting but rolled differently to obtain "extreme structures". Test pieces were taken at mid-thickness and at quarter thickness of each plate, and were

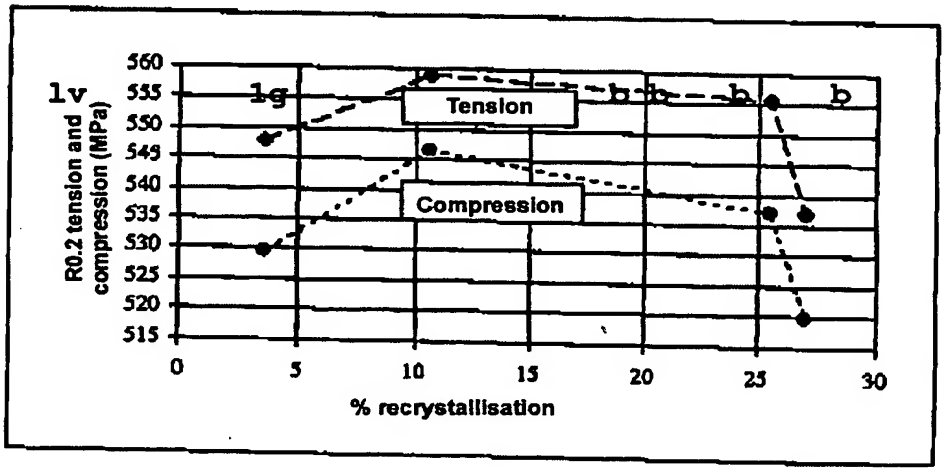
requenched, tensioned after 10 hours of aging, and annealed to T651 at the CRV to give an identical finishing in all cases. The values of $R_{0.2}^{tens}(L)$ and $R_{0.2}^{comp}(L)$ obtained for these plates are summarised in table 2 and in Figure 2. The recrystallisation ratios indicated were measured on the end of a tension test piece. It was found that:

- the values of $R_{0.2}^{tens}(L)$ are systematically higher than the values of $R_{0.2}^{comp}(L)$, regardless of the recrystallised fraction;
- there is no clear relation between the recrystallised fraction and tension - compression asymmetry.

"Therefore, it is probable that the recrystallised fraction does not provide a sufficient description of differences in microstructure causing a difference in the asymmetry of yield stresses between sections and plates.

Table 2. Yield stresses (L) for 7010 - T651 as a function of recrystallisation ratio

Alloy	Plate (recryst.)	Direc-tion	Location	% rex. CRV	% tension after quenching	R0.2 MPa	Rm MPa	A%	R0.2 comp. MPa	DR0.2 MPa
7010-01	451013 (thin)	L	1/4 t	3.6	2%	548	572	7.1	529	19
			1/2 t	10.6	"	559	598	7 6	546	13
	451010 (thick)	L	1/4 t	27	2%	537	561	6.8	520	17
			1/2 t	25.5	"	556	594	7.2	537	19



1 b

Figure 2. Yield stresses for 70 mm thick 7010 - T651 plates as a function of the recrystallisation ratio. See discussion in text.

pages 14, 15 (including table 3 and figure 3), 16 (including figure 4)

REDACTED

(REDACTED)

3.3. Influence of ~~annealing~~ ageing

We have already noted that asymmetry between tension and compression in plates is lower when ~~annealing~~ ageing is more complete (see figure 6 derived from [3]). Note that this observation is valid for the L and TL directions, and that most of the asymmetry reduction occurs between the T651 and T7951 tempers.

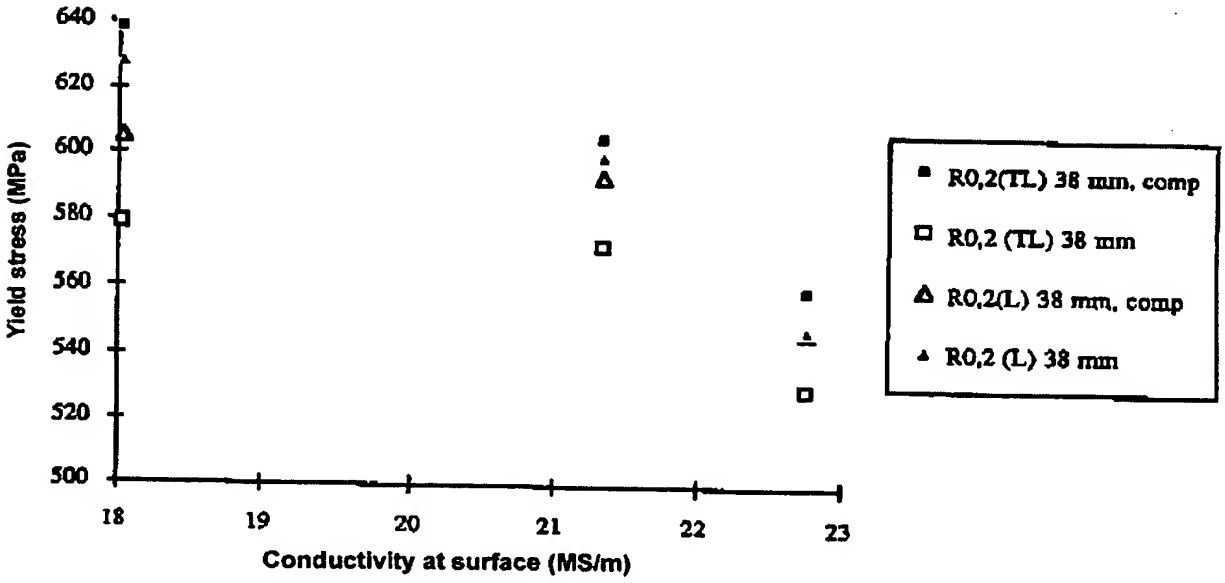


Figure 6. Compromise between yield stress and conductivity for a 38 mm thick 7449 plate. See text

We carried out tests on two 38 mm thick plates to determine the influence of the ~~annealing~~ ageing cycle (results obtained by [redacted] in Issoire, and presented in Appendix 1) in order to identify the effect of an increase in the ~~annealing~~ ageing temperature for a constant equivalent time corresponding approximately to the ~~annealing~~ ageing peak. The four chosen ~~annealings~~ ageing practices were:

[3] Note CRV/ALT/TJW.144 by T. Warner "Analysis of thick DTP 7449 plates".

- 48 h at 120°C, the conventional T651;
- 67 h at 120°C, corresponding to the "true" ~~annealing~~ ageing peak for $R_{0.2}^{tens}$(L) according to our previous results (see e.g. [4]);
- 9h at 140°C, ~~annealing~~ ageing to the annealing peak for $R_{0.2}^{tens}$(L) at 140°C calculated by equivalent time;
- 3h at 150°C, ~~annealing~~ ageing to the annealing peak for $R_{0.2}^{tens}$(L) at 150°C calculated by equivalent time.

Variations to the yield stress - conductivity compromise are given in figure 7. Note that ~~annealing~~ ageing for 67 h at 120°C gives better yield stresses in tension and in compression. Furthermore, note that asymmetries in the yield stress reduce apparently monotonously as ~~annealing~~ ageing continues (see figure 8).

[4] Note CRV/MA-MAR/TJW.96.514 dated 23/11/96 "Laboratory tests for T7751 treatments on 7449".

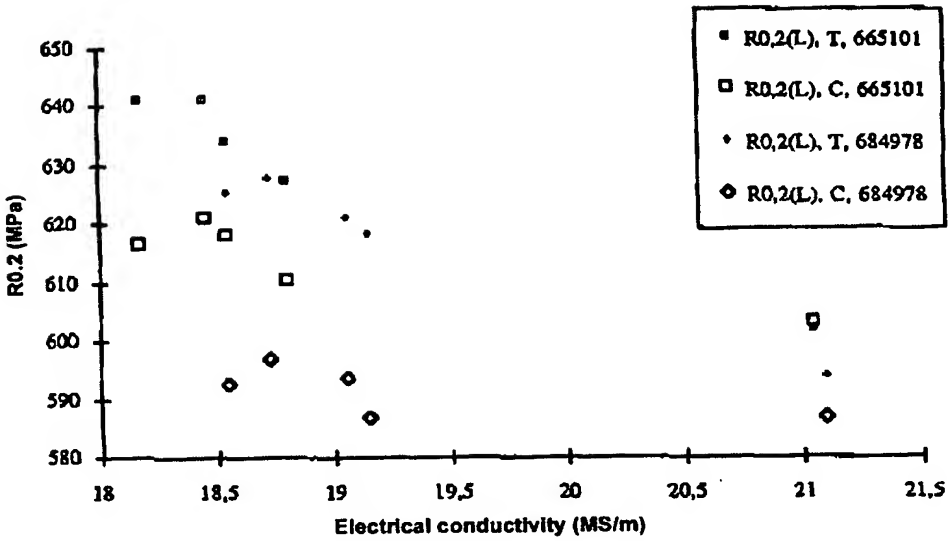
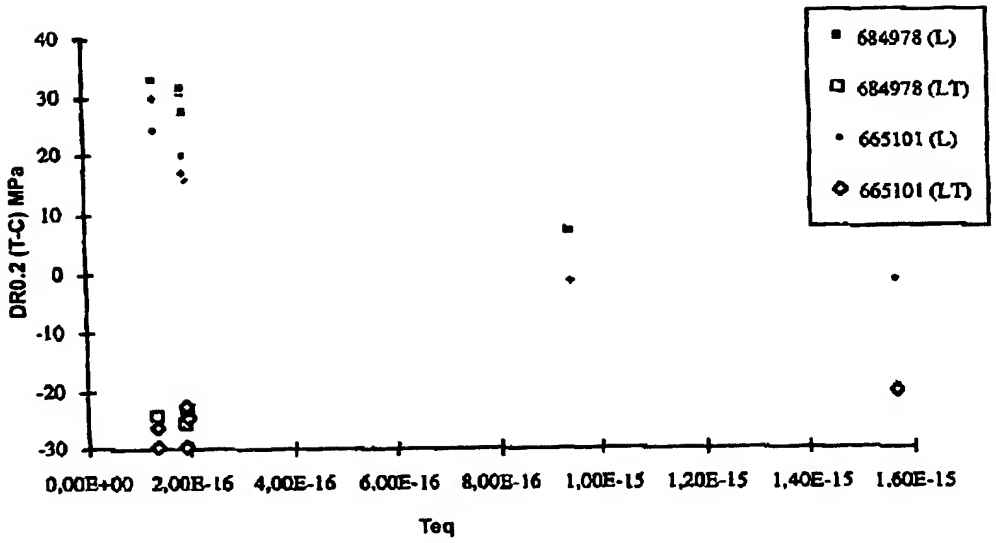


Figure 7. Graphic view of the compromise between yield stress and conductivity obtained for different degrees of annealing at the peak and for T7951 ~~annealing~~ aging on the same plates (complete data in Appendix



1).

Figure 8. Yield stress asymmetries as a function of equivalent ~~annealing~~ ageing time for data in Appendix 1.

3.4. Alloy composition: solid solution and second phases

No specific test was carried out to study this parameter of the problem. The only element used to make a comparison of the different compositions is taken from the experimental plan that contributed to development of the NAV (for example see report [5]). In this case, 22 mm thick plates derived from 380 x 120 mm formats cast at the CRV, were rolled, quenched and tensioned directly (a few minutes waiting) before ~~annealing~~ ageing to the peak (between 24 and 96 h at 120°C). The first observation is relatively low differences

[5] P. Gomiéro et al (1995) CRV report No. 3715 "New very high strength 7xxx alloy for wing".

between tension and compression (see figure 9 below), and also a significant trend (at $\alpha = 0.01$) to increase asymmetry of the yield stress with oversaturation of the alloy. In figure 9 below, this oversaturation is shown by the quantities of insoluble phases calculated by Pro_phase for each alloy.

It will be noted that the fraction by volume of phases not in solution has a minimum value ($\approx 0.5\%$) corresponding to phases with Fe linked to the purity of the base. Moreover, the threshold value is equal to about -13 MPa, which corresponds to the inherent difference in determination of conventional yield stresses [redacted]

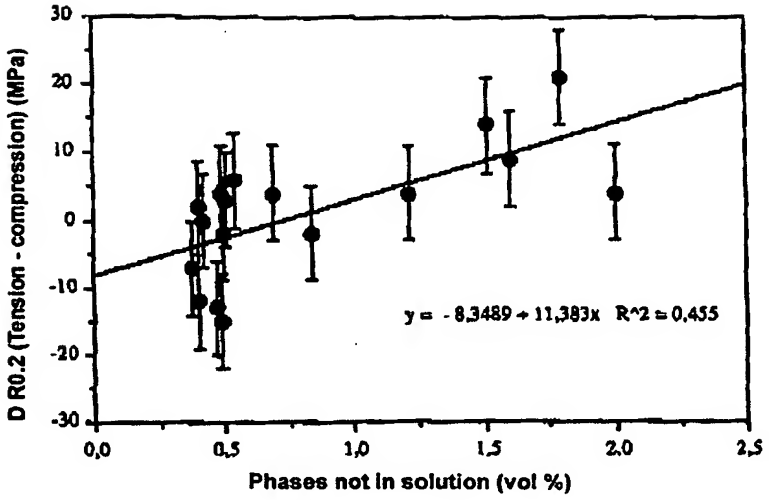


Figure 9. Yield stress asymmetries in the longitudinal direction as a function of the fraction by volume of phases not in solution (pro_phase calculations) for 22 mm thick plates corresponding to the experience plan used for development of the NAV (7 x 49).

4. Use of industrial manufacturing properties

(REDACTED)

pages 24 and 25

(including figures 10 and 11)

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5. Discussion

The information in §3 and 4 can be summarised as follows. The value of $R_{0.2}^{tens}(L) - R_{0.2}^{comp}(L)$ will reduce as:

- the equivalent ~~annealing~~ ageing time increases;
- dissolution of the alloy improves;
- the ~~tension ratio~~ permanent set reduces (temper T651).

The granular structure also has an influence (see §3.1), but the important parameter is not only the local recrystallisation ratio. Furthermore, note in particular that the compression curves of plates demonstrate a significantly higher micro-plasticity than in tension.

These observations can be rationalised by our proposal of a subjacent mechanism based on the development of micro-stresses during the stress relaxation operation. The softest parts of the microstructure (for example recrystallised grains) become plastic earlier than the hardest parts, and are thus once again in compression after the tension operation (see diagram figure 12). If the product is loaded once again in tension, there will be a marked initiating plasticity at a high stress. On the other hand, if the product is loaded in compression, the softest parts of the microstructure, which are themselves already in compression, will start to creep

at a lower stress than in tension. Therefore, the result will be firstly a more rounded curve in compression corresponding to greater micro-plasticity, and also a lower yield stress. This asymmetry will continue until T651 ~~annealing~~ aged, but will reduce for tempers closer to over-~~annealed~~ aged tempers due to thermal relaxation of microstresses. Therefore, the asymmetry between tension and compression $R_{0.2}$ can be explained by **a remanent Bauschinger effect.**

The generation of residual microstresses during preliminary tension has been studied many times in the external bibliography, particularly concerning two-phase model materials (for example see [6]). Il has been demonstrated that the difference in creep stress thus generated for model materials in the pre-tension direction was approximately:

$$\Delta\sigma_{Bausch} = 2.\sigma_{res} \qquad \text{Eq. 4}$$

$$= 2.f.\mu.\varepsilon_p^* \qquad \text{Eq. 5}$$

where σ_{res} corresponds to the average stress generated during tension in the softest part of the material, f is the fraction by volume of hard particles, μ is the shear modulus, and ε_p^* is a parameter representing plastic pre-deformation and intrinsic characteristics of the material.

[6] e.g. L.M. Brown and W.M. Stobbs (1971) in Phil Mag 23, pp. 1185 and 1201.

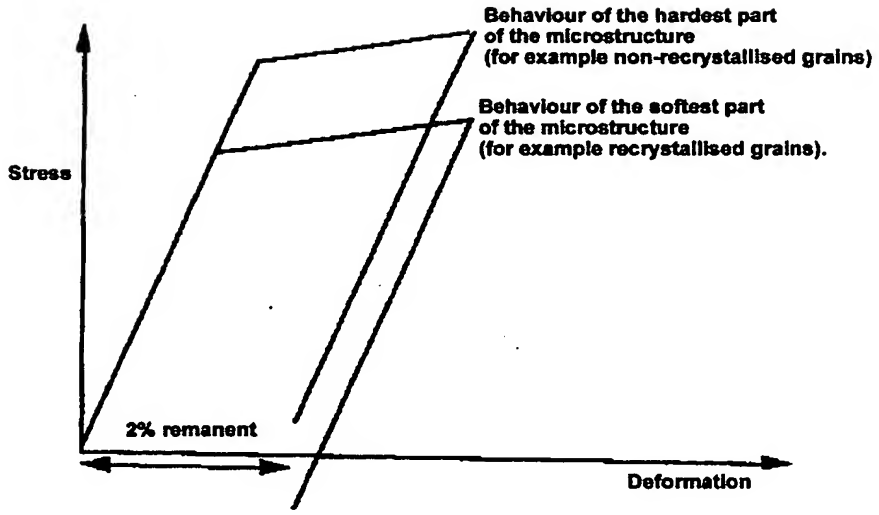


Figure 12. Principle diagram for generation of microstresses during tension of thick plates.

This model of a two-phase material can be extrapolated to suggest that the development of micro-stresses would be higher if the local hardness of the product is heterogeneous and high, and if the pre-tension is high. Therefore, the following factors would generate micro-stresses that have an adverse effect on $R_{0.2}^{comp}$ (L):

- a local heterogeneity of the granular structure (recrystallisation ratio or presence of second phases in the specific case);
- long aging (increase in the mechanical characteristics of the different parts of the structure and thus their capacity to generate stresses);
- a high tension ratio.

The microstresses thus generated are affected by a diffusional relaxation during ~~annealing~~ ageing, so that they

can be reduced and thus also reduce the tension - compression asymmetry. This effect would explain the variation of the tension - compression asymmetry with ~~annealing~~ ageing, since it is shown in the bibliography[7] that the rate of stress relaxation is defined as follows:

$$\frac{\sigma_{res}(t,T)}{\sigma_{res}^{0}} = \exp\left(-Ct_{eq}^{m}\right) \qquad \text{Eq. 6}$$

Where σ_{res}^{0} is the residual stress at the beginning of heat treatment, C and m are constants that depend on the material, and t_{eq} (= t.exp(-Q/RT)) is an equivalent time parameter for the relaxation mechanism that takes place. The activation energy corresponding to the stress relaxation would be the auto-diffusion activation energy of aluminium for average temperatures, which is approximately the same as the value that we use for calculating the equivalent ~~annealing~~ ageing time.

[7] H. Holzapfel, V. Schulze and O. Vöhringer (1994) in "Shot Peening" proceedings, Strasbourg, 22/09/94, p. 15 - 30.

Equations 2, 3 and 6 can be combined to represent a theoretical asymmetry of yield stresses:

$$R_{0.2}^{tens}\ (L) - R_{0.2}^{comp}(L) = 0.015\ R_{0.2} - 10^{-4} \left(\frac{d\sigma}{d\varepsilon}\right)_{0.2\%} + 2\ \sigma_{res}^{0} \exp\ (- Ct_{eq}^{m})$$

$$\approx - 12 + 2\ \sigma_{res}^{0} \exp\ (- Ct_{eq}^{m})$$

The shape of this theoretical curve (eq. 7) is compared below (figure 13) with data in figure 6 for the 665101 and 684978 plates by adjusting the values of σ_{res}^{0}, C and m (see legend in the figure). The shape of the variation of the asymmetry of the yield stress is correctly shown with physically reasonable values of σ_{res}^{0}, C and m (see legend in the figure).

One advantage of this model is that it implies that there is probably a compression yield stress peak that is more over~~annealed~~ aged than the tension peak. A first convolution of the above calculations with our knowledge about the rate of ~~annealing~~ aging of 7449-01 (for example see [4]) suggests that the ~~annealing~~ aging peak for $R_{0.2}^{comp}(L)$ for a thick plate is located at about 24 h at 135°C ($t_{eq} = 3.4 \times 10^{-16}$).

This assumption of a remanent Bauschinger effect could also explain other qualitative aspects of our observations:

- the fact that $R_{0.2}^{comp}$ (L) of Tx511 tempers is higher than $R_{0.2}^{tens}$ (L) would be due to the homogeneity of the microstructure and the resulting lack of generated micro-stresses. Therefore, the observed difference would only be the difference corresponding to the geometric effects of the use of conventional yield stresses (see §2);

- the fact that the difference between the tension and compression is not as high for 7150 - T651 would be due to the greater ~~annealing~~ ageing performed for 7150 - T651 than for a 7449 - T651.

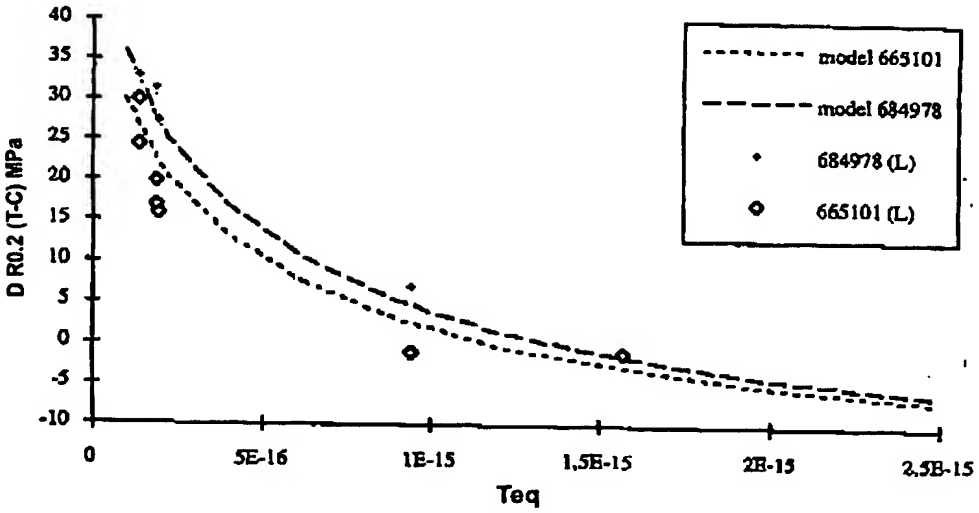


Figure 13. Comparison of asymmetries of $R_{0.2}$ as a function of the ~~annealing~~ ageing t_{eq} and calculations using equation 6 (σ^0_{res}, $C=5.10^7$, $m=0.5$, $\sigma^0_{res}=40$ MPa (684978) or 35 MPa (665101) respectively).

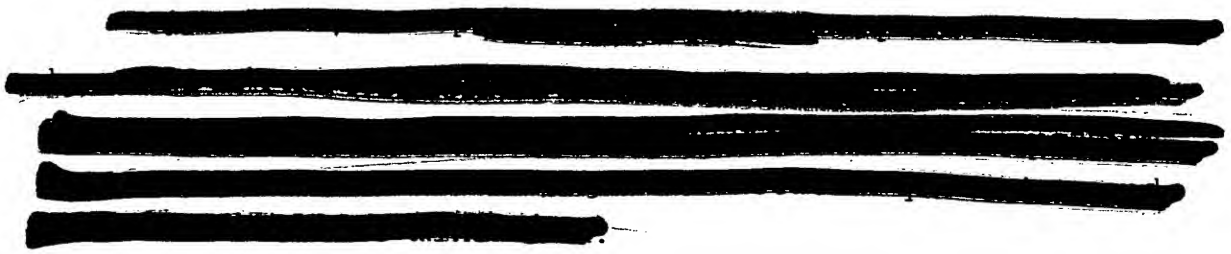
6. Conclusions

According to the results given above, the value of $R^{tens}_{0.2}$ (L) - $R^{comp}_{0.2}$ (L) will reduce as:

[redacted]

- the equivalent ~~annealing~~ ageing time increases;
- dissolution of the alloy improves;
- the ~~tension ratio~~ permanent set reduces (temper T651).

The granular structure also has an influence (see §3.1), but the important parameter is not only the local recrystallisation ratio. [redacted]



These observations are rationalised in §5. It is proposed that the asymmetry of yield stresses is due to a remanent Bauschinger effect due to the development of microstresses during stress relaxation by tension, this effect reducing during ~~annealing~~ ageing due to relaxation of these microstresses. Note that there is an inherent asymmetry in the method of measuring the yield stress implying a negative $R_{0.2}^{tens}(L) - R_{0.2}^{comp}(L)$ difference if there is no difference between the rational tension and compression curves.

Appendix 1. Influence of the ~~annealing~~ ageing temperature on tension and compression yield stresses: data given by [redacted]

ANNEALING RATE T651 on 7449 - T = 38 mm

PEAK ~~ANNEALING~~ AGEING FOR STUDY OF THE INFLUENCE OF THE ~~ANNEALING~~ AGEING TEMPERATURE ON $R_{0.2}$ IN COMPRESSION

		Tension						Compression				K1C	
		LL direction			TL direction			LL direction		TL direction		C725	
Annealing	Plate No.	Rm(MPa)	Rp0.2(MPa)	A5.65(%)	Rm(MPa)	Rp0.2(MPa)	A5.65(%)	Rp0.2(MPa)	E(MPa)	Rp0.2(MPa)	E(MPa)	L/T	T/L
46h At 120°C	665141	672	642	14.6	669	631	11.5	617	71948	661	75090		
		671	640	14.6	669	630	11.6	616	72802	659	75459		
		Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:		
		672	641	14.6	669	631	11.6	617	72376	660	75726		
	684978	660	626	11.6	656	614	12.9	592	76876	638	75636		
		660	626	11.7	656	614	12.8	593	72802	638	76945		
		Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:		
		660	626	11.7	656	614	12.6	593	74839	638	75991		
67h At 20°C	665141	670	641	14.0	669	631	14.6	621	75408	661	75636		
		670	641	9.9	668	632	11.3	621	75105	661	75459		
		Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:		
		670	641	12.0	669	632	12.8	621	76257	661	75548		
	684978	661	628	14.8	656	615	11.2	597	73068	641	75636		
		661	629	10.8	655	615	11.9	597	75090	640	75813		
		Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:		
		661	629	14.8	656	615	11.6	597	74529	641	75726		
9h At140°C	665141	663	634	14.1	661	627	14.5	620	74751	650	72625		
		662	634	14.3	661	627	14.7	616	75813	653	75813		
		Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:		
		662	634	14.2	661	627	14.6	618	76252	652	74219		
	684978	653	621	11.1	649	612	11.1	593	74696	636	73742		
		652	621	14.1	648	612	14.5	594	73034	634	74219		
		Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:		
		653	621	12.6	649	612	12.8	594	73886	635	73981		
5h At150°C	665141	656	628	14.2	655	621	14.2	610	71269	645	72477		
		656	627	14.8	655	621	14.6	611	72656	642	73312		
		Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:		
		656	628	14.5	655	621	14.4	611	72063	644	73396		
	684978	651	619	14.6	647	614	14.9	586	72979	638	72802		
		649	618	14.9	649	611	11.0	588	72802	638	74396		
		Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:	Moy.:		
		650	619	14.8	648	613	13.0	587	72991	638	73599		

Appendix 2. Industrial tests, effect of [redacted]

pages 37 and 38

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Appendix 3. Analysis of $R_{0.2}(L)$ in compression for industrial plates [redacted]

Set of compression values on 7449-7851

t (mm)	Plate No.	RHU/IS tests				CEAT tests				Quality report values					
		R0.2 L	E L	R0.2 TL	E TL	R0.2 L	E L	R0.2 TL	E TL	R0.2 L	E L	R0.2 TL	E TL	Note	R0.2 L
19	684982	579	72300												
18	684982	579	72462												
38	685101	613	73814	631	74042										
38	685101	615	74188	634	75210					614	74880	653	74880	(Average RHU/IS)	613
38	685101														615
38	685101					609	74000	647	75200	599	73980	647	74500	Average 1st CEAT test	599
38	685101					608	73700	647	74780						598
38	685101					591	73980	647	74700						601
38	685101					612	74180	656	75800	614	73800	655	74900	Average 2nd CEAT test	612
38	685101					616	73100	655	74700						615
38	685101					616	73600	656	74600						616
38	684978	584	71147								71150				
38	684978	588	71973								71200			(Report error: 71950)	
38	684983	584	71798												
38	684983	580	71973												
38	684983	580	72816												
38	684983	580	71361												
38	684983	588	72883												
38	684983	594	72564							593	72400				
										594	72560				
60	654148	575	73918	631	74398										
60	654148	578	74068	627	74149										
60	654148	578	70872												
60	654148	578	72214							578	71000				
										578	72808				
60	684978			633	72334	588	73600	633	75600	594	73700	653	74800		
60	684978			631	72504	602	73600	631	74800					CEAT average	
60	684978					595	73600	635	74800						
60	684977	577	71833												
60	684977	581	71324												
38	684980	581	71973												
38	684980	581	72016							591	72600				
38	684980	590	72816							591	72800				
38	684980	590	72050												
38	684980	587	71028												
38	684980	588	72283												
60	654145	561	71973	622	74260										
60	654145	568	72748	620	74219										
15	684984	581	71977												
15	684984	579	71324												
15	684981	583	72050												
15	684981	586	72207												
15	684979	573	72160												
15	684979	578	72030												
15	685102	600	73936	648	73688					600	72600	648	73400		600
15	685102	602	73156	648	73156					602		648		EC = average RHU/IS	602
	Moy	585.56	72269	636.50	73901	589.22	73700	645.11	74811	594.83	72488	647.33	74900		607.10
	Etype	11.31	891	12.59	898	13.23	300	9.83	309	11.86	1048	7.71	824		7.64

pages 41 and 42

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EXHIBIT B

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CRV/ALT/TJW

Your Ref.:

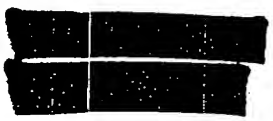
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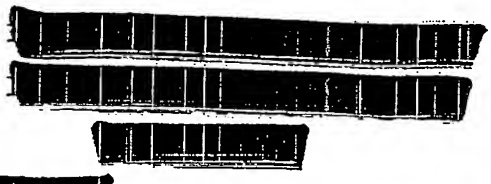
Annealing peak for compression yield stress of 7449 plates: a gain of 25 MPa compared with the existing T6

Author:
T. WARNER
F. CARIONI

To:



For info:





Signature:

Project or service No.: 4537
Requested by: Rhenalu

Introduction

Previous work[1] related to the asymmetry between tension and compression yield stresses identified a role of residual micro-stresses on the compression yield stress in the L ($R_{0.2}^{comp}$(L)) direction of plates made of 7449. This reasoning suggested that an annealing peak should be provided for $R_{0.2}^{comp}$(L) at annealing equivalent to 24 h at 135°C, in other words half way between existing T651 and T7951 annealing. The objective of this work is to check this reasoning, and if applicable to evaluate the gain in $R_{0.2}^{comp}$(L) thus obtained. This note reports experimental results obtained in this study. It will be followed by a more complete report analysing the variation of the shape of compression curves and mechanisms subjacent to the observed phenomena.

[1] Note CRV/ALT/TJW 97.362

Methods

Samples of two 7449 plates in the T351 temper were used for these tests: one 25 mm thick plate (708693), one 38 mm thick plate (684978) derived from the same casting #25 (see composition in table 1 below). The relevant parameters for the procedure for the two plates are indicated in table 2.

Table 1. Composition of plates used for this study

Casting	Si	Fe	Cu	Mg	Zn	Zr
99381	0.039	0.063	1.96	2.15	8.98	0.109

Table 2. Parameters for the procedure for the two plates

Procedure parameter	Plate 684978	Plate 708693
Widening	1100 mm -> 2500 mm	1100 mm -> 2500 mm
End of hot rolling T°	390	378
T° MeS	474°C	475°C
Quenching-tension waiting time	10.5 h	0.75 h
Tension	2.4%	2.8%

Eight annealing operations varying from the existing T651 to T7951 were carried out on laboratory test piece blanks taken at mid-thickness of the two plates (see table 3). The rise gradients between levels were fixed at 16°C/h to simulate industrial treatment. At the end of annealing, the test pieces were taken from the furnace and cooled in Kerlane. In each case, we measured R (L) (3 Æ13 mm test pieces, L = 25 mm) and R (L) (2 TOR 6 test pieces) on test pieces taken from the mid-thickness plates, and the conductivity on a test piece taken from the surface.

Table 3. Annealings used in this study

Note	Annealing	Teq
T651	48h à 120°C	1.4×10^{-16}
T651 (135°C)	12h à 135°C	1.4×10^{-16}
	16h à 135°C	2.9×10^{-16}
Calculated "compression peak"	24h à 135°C	3.0×10^{-16}
	30h à 135°C	3.8×10^{-16}
	24h à 120°C + 5.5h à 150°C	3.8×10^{-16}
	24h à 120°C + 11h à 150°C	6.5×10^{-16}
T7951	24h à 120°C + 17h à 150°C	9.4×10^{-16}

Calculated annealing operations for increasing T° at 16°C/h, reducing T° at -65°C/h, Q/R = 15866 K^{-1}.

Results

The values of the compression and tension yield stress are given in tables A1 and A2 in the appendix. Figures 1 and 2 show a graphic view. It is found that:

- the properties obtained on these plates after T7951 annealing are consistent with the properties obtained during the qualification;
- the annealing peak for $R_{0.2}^{comp}$ (L) is located at t_{eq} equal to about 4 x 10^{-16} (for example 30 h at 135°C) for the two plates;
- for this annealing, variations of the property compared with classical T7951 and T651 used are about:

+20 to +25 MPa in $R_{0.2}^{comp}$ (L) compared with T651 and T7951

-5 to +3 MPa (or +24 to +38 MPa) in $R_{0.2}^{tens}$ (L) compared with T651 (and T7951)

-12 to -13 MPa (or +25 to +30 MPa) in $R_{0.2}^{tens}$ (L) compared with T651 (and T7951)

Conclusions

As expected, the ~~annealing~~ ageing peak for the compression yield stress is between the T651 and T7951 annealings. The equivalent ~~annealing~~ ageing time necessary to reach this peak is about 4 x 10^{-16}h, namely for example ~~annealing~~ ageing of 30h at 135°C (compared with calculated 24 h at 135°C). The improvement in static properties compared

with existing ~~annealings~~ ageing treatments is probably useable. Furthermore, ~~annealing~~ ageing done at low temperature could reduce the decoration of grain boundaries during ~~annealing~~ ageing and thus improve the resistance to exfoliating corrosion of the product and toughness for a given annealing t_{eq}.

Recommendations

1. Perform a more complete characterisation of the compromise of properties that can be achieved with this new ~~annealing~~ ageing, on at least two plates. The objective is to evaluate:

- static properties;
- resistance of exfoliating and stress corrosion;
- the resistance to fatigue on a test piece with hole;
- toughness (R curve, Boeing conditions for 7055-T7751?)

2. Present this new compromise of properties to aircraft manufacturers.

Figure 1: Annealing rate 25 mm 708693

Note: Error bars correspond to the 95% confidence interval

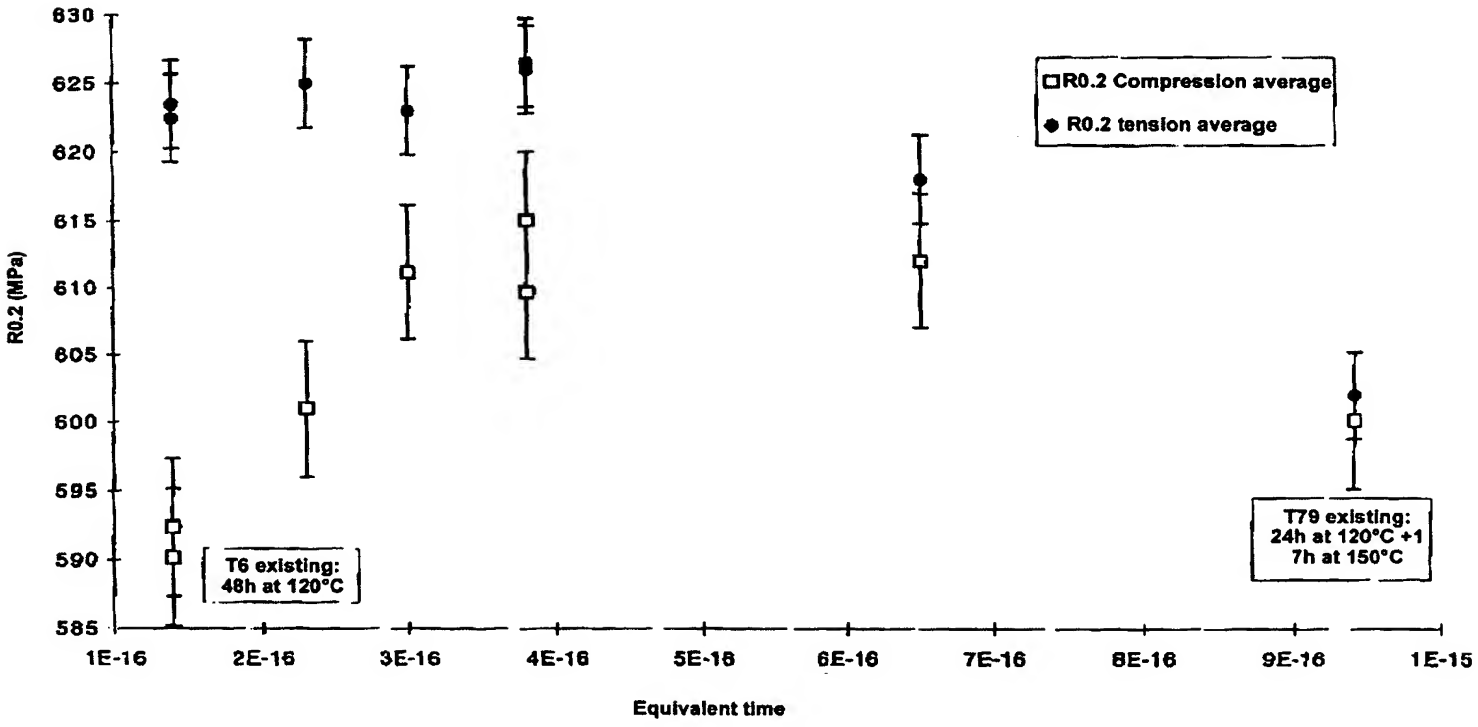


Figure 2: Annealing rate 38 mm 684978

Note: Error bars correspond to the 95% confidence interval

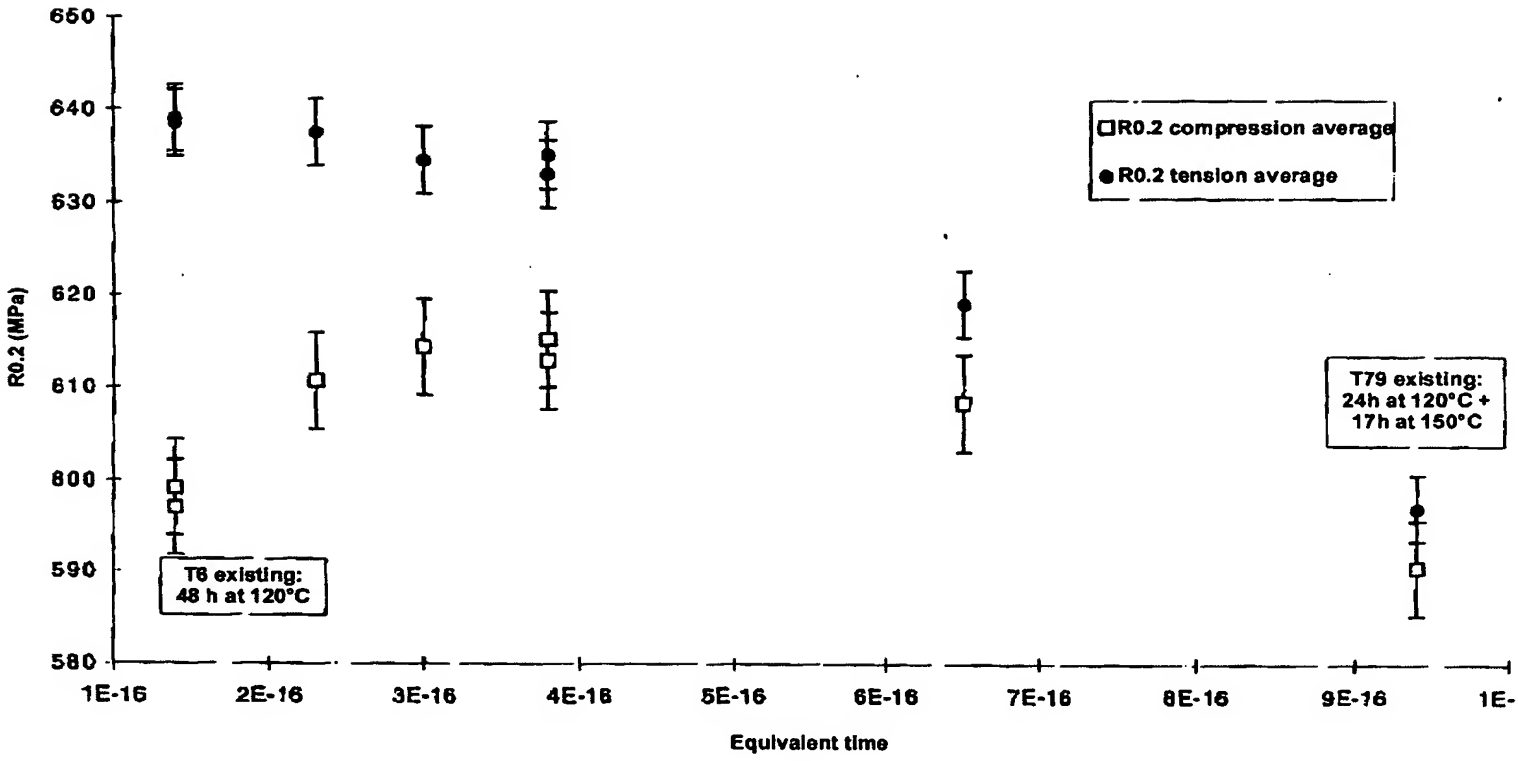


Table A1. Results of mechanical properties (tension and compression) and electrical conductivities

Plate mark	Annealing T Th	Tension R0.2 (MPa)	Tension Rm (MPa)	Tension A %	Compression R0.2 (MPa)	Equivalent T TH (10^-16 h)	Elect. Conductivity MS/s
708693 t = 25 mm	48 h 120°C	626 621	667 665	13.6 12.9	593 588 590		
	Average Standard deviation	623.5 3.5	666.0 1.4	13.3 0.4	590.2 2.6	1.4	18.65
708693 t = 25 mm	12 h 135°C	623 622	664 663	12.4 14.3	595 587 586		
	Average Standard deviation	622.5 0.7	663.5 0.7	13.3 1.4	592.3 5.1	1.4	18.65
708693 t = 25 mm	18 h 135°C	626 624	661 658	14.0 11.7	600 603 600		
	Average Standard deviation	625.0 1.4	659.5 2.1	12.9 1.6	601.0 1.7	2.9	19.2
708693 t = 25 mm	24 h 135°C	623 623	664 662	11.6 12.9	614 609 611		
	Average Standard deviation	623.0 0.0	663.0 1.4	12.3 0.9	611.2 2.8	3.0	19.8
708693 t = 25 mm	30 h 135°C	625 628	653 656	11.1 11.7	608 611 611		
	Average Standard deviation	626.5 2.1	654.5 2.1	11.4 0.5	609.7 1.9	3.8	20.1
708693 t = 25 mm	24 h 120°C + 5.5 h 150°C	625 627	653 655	12.4 10.7	611 618 617		
	Average Standard deviation	626.0 1.4	654.0 1.4	11.6 1.2	615.0 3.9	3.8	20.1
708693 t = 25 mm	24 h 120°C + 11 h 150°C	618 618	643 642	10.7 11.3	614 616 607		
	Average Standard deviation	618.0 0.0	642.5 0.7	11.0 0.4	612.0 4.9	6.5	21.1
708693 t = 25 mm	24 h 120°C + 17 h 150°C	602 602	631 630	11.7 12.3	601 604 597		
	Average Standard deviation	602.0 0.0	630.5 0.7	12.0 0.5	600.2 3.5	9.4	21.8
T7951	24 h 120°C + 17 h 150°C	599	629	11.1	602	9.4	21.65

Table 3. Summary results of mechanical characteristics (tension and compression) and electrical conductivities

Plate mark	Annealing T Th	Tension R0.2 (MPa)	Tension Rm (MPa)	Tension A %	Compression R0.2 (MPa)	Equivalent T TH (10^-16 h)	Elect. Conductivity MS/m
708693. T 25mm	48 h 120°C	623.5	668.0	13.3	590.2	1.4	18.65
"	12 h 135°C	622.5	663.5	13.3	592.3	1.4	18.85
"	18 h 135°C	625.0	659.5	12.9	601.0	2.3	19.20
"	24 h 135°C	623.0	653.0	12.3	611.2	3.0	19.60
"	30 h 135°C	626.5	654.5	11.4	609.7	3.8	20.10
"	24 h 120°C + 5.5 h 150°C	626.0	654.0	11.6	615.0	3.8	20.10
"	24 h 120°C + 11 h 150°C	618.0	642.5	11.0	612.0	6.5	21.10
"	24 h 120°C + 17 h 150°C	602.0	630.5	12.0	600.2	9.4	21.60
Industrial result T7951	24 h 120°C + 17 h 150°C	599.0	629.0	11.2	602.0	9.4	21.65
684978. T 38mm	48 h 120°C	638.5	676.0	12.4	596.3	1.4	18.40
"	12 h 135°C	639.0	673.0	11.8	599.2	1.4	18.75
"	18 h 135°C	637.5	668.0	12.0	610.7	2.3	19.15
"	24 h 135°C	634.5	663.5	11.0	614.3	3.0	19.75
"	30 h 135°C	639.0	663.5	10.5	615.2	3.8	20.00
"	24 h 120°C + 5.5 h 150°C	635.0	662.0	11.2	612.8	3.8	20.15
"	24 h 120°C + 11 h 150°C	619.0	646.0	10.5	608.3	6.5	21.25
"	24 h 120°C + 17 h 150°C	597.0	631.0	10.7	590.5	9.4	21.90
Industrial result T7951	24 h 120°C + 17 h 150°C	594.0	625.0	9.2	587.0	9.4	21.95

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EXHIBIT C

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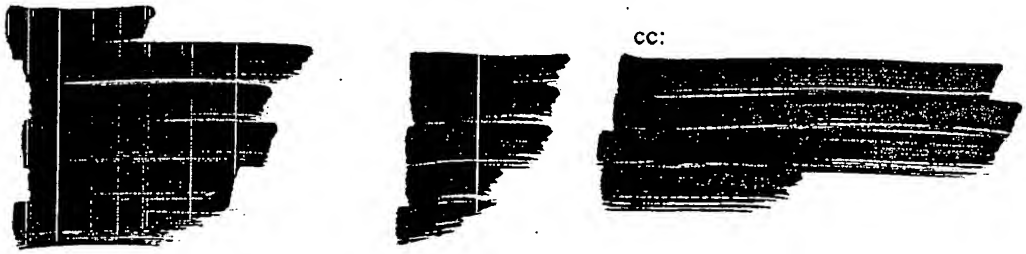
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Development of an improved CYS temper for 7449 for UWS of Boeing aircraft – current status

Authors:
T. Warner
D. Aumay

Signature :



cc:

Project: 5162700 (scénario 0309 - 430)

Customer: RHU (ATI)

Summary

The aims of the current work were:
- to complete the characterization of the T7951 temper with respect to [redacted]s goals;
- to assess a T6x51 temper (intermediate between current T651 and T7951) with respect to these same goals, and to optimize this temper if appropriate.

These results indicate that 7449-T6x51 would meet [redacted]CYS(L) requirement. However, it would not achieve the EXCO target, and probably not the toughness target (see table 4.1 reproduced below). 7449-T7951 plate meets the EXCO target but is currently insufficient in both CYS(L) (by about 18 MPa) and K_{app}(L-T) (by about 15MPa$\sqrt{m}$).

Comparison of properties of T6x51, T7951 tempers with [redacted] goals (typical values)

Property	Property goal (SI)	7449-T6x51	7449-T7951 properties
CYS(L) T/2 sampling	613 MPa	610 MPa	595 MPa
K_{app}(L-T) T/2, B = 6.35 mm, W = 406 mm	101 MPa$\sqrt{m}$	?	84.5 MPa$\sqrt{m}$
Exfoliation corrosion (ASTM G34)	EB	EC	EB

Further conclusions of this study include the following:

- for the T6x51-type tempers there are no clear variations of CYS(L) with the temperature of the first aging step, nor with the temperature of the second aging step for a given equivalent aging time (see figure 3.1);
- the performance of a third aging step (24h @ 120°C) increases CYS(L) significantly for both plates (see figure 3.1);
- none of the modifications in the T6x51 aging treatment showed an improvement in EXCO performance;
- the SCC performance of 7449-T6x51 is inferior to that of 7449-T7951 as expected (see figure 3.2). However, there remains an unexplained inconsistency between these recent results and those obtained previously (see figure 3.3).

[redacted]

(REDACTED)

Recommendations

1. Assess the K_{app}(L-T) of 7449-T6x51 plate in order to complete the property comparison (need plate from Issoire!).
2. Evaluate the potential of a third aging step for a temper nearer T7951: this study would have to be preceded by a clear assessment of the IP position.
3. Evaluate the property balance (K_{app}, CYS(L), EXCO) achieved with a high purity product (see recent Issoire cast), in the following tempers: T7951, T6x51, T7651.
4. Roll a plate (if necessary from an Issoire ingot) at Ravenswood, in order to assess the impact of the higher HR temperatures achievable with a more powerful mill.
5. Identify the origin of the differences between initial DCB data and subsequent results (testing procedure, ingot size, plate thickness, ...).

1. Introduction

The goals of this memo are to summarize current status on the development of an upper wing skin (UWS) alloy for Boeing, and to recommend directions for future work. Current property goals as communicated by [redacted] are indicated in table 1.1; also in this table, typical properties of 7449-T7951 are presented. 7449-T7951 does not meet the CYS(L) goals, but displays adequate EXCO performance. Evaluation of its toughness was one of the goals of this phase (see table 3.3).

We had previously demonstrated that the peak CYS(L) of 7449 is achieved for an aging treatment between our current T651 (defined with respect to TYS(L) and UTS(L)) and T7951 (a slight overage). This effect is illustrated in figure 1.1, abstracted from reference[1]. Also in this note, it was postulated that a rapidly quenched product aged at low temperatures (< 140°C approximately) should show good EXCO resistance, though not good SCC performance (see for example [2] for the underlying reasoning).

Table 1.1. [redacted] property goals.

Property	Property goal (US)	Property goal (SI)	7449-T7951 properties
CYS(L) T/2 sampling	B-value of 87 ksi, i.e. typical ≈ 89 ksi	B-value 599 MPa, i.e. typical ≈ 613 MPa	Typical 595 MPa
K_{app}(L-T) T/2, B = 6.35 mm, W = 406 mm	92 ksi√in (typical)	101 MPa√m (typical)	(see table 3.3)
Exfoliation corrosion (ASTM G34)	EC minimum (EB typical for comfort)	EC minimum (EB typical for comfort)	EB typical

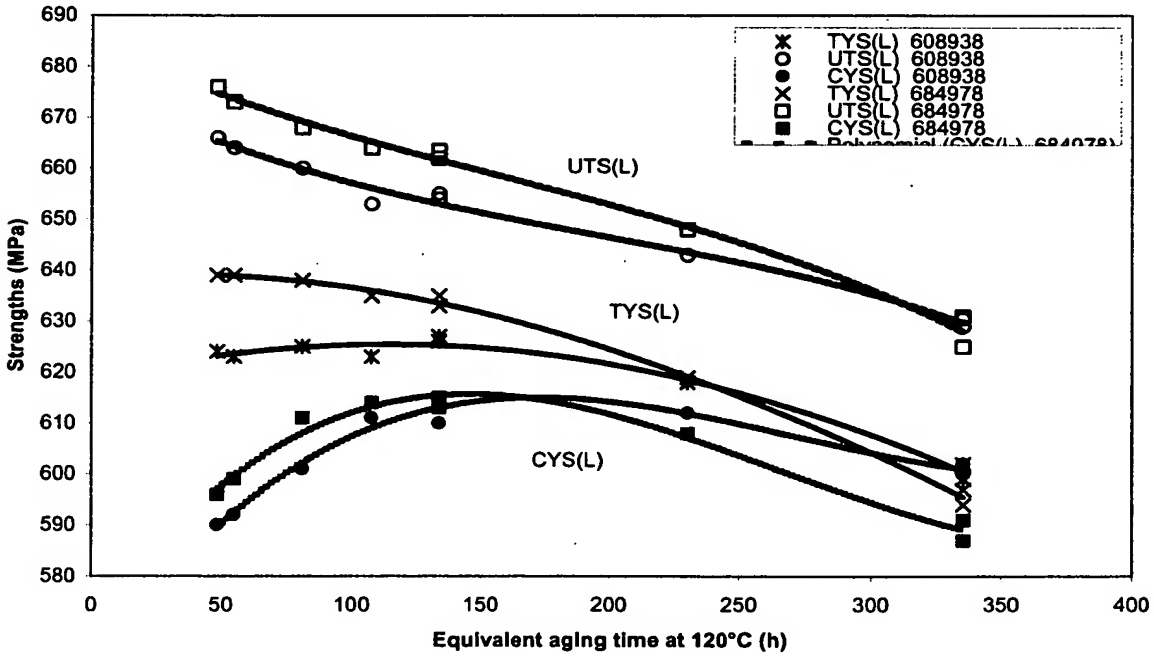


Figure 1.1. Identification of peak aging conditions for CYS(L) of 7449 (abstracted from CRV/ALT/TJW 98.515). Two plates were tested (608935, 23.5 mm thickness and 684978, 38 mm thickness). Current T651 and T7951 aging treatments are indicated.

The aims of the current work were thus:

- to complete the characterization of the T7951 temper with respect to [redacted]'s goals;
- to assess a T6x51 temper (intermediate between current T651 and T7951) with respect to these same goals, and to optimize this temper if appropriate.

It should be noted that our original intention was to assess both T7951 and T6x51 in K_{app}, but that we were unable to source sufficient 1" plate in a T351 or TAF temper to evaluate both tempers.

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[1] CRV/ALT/TJW 98.515

[2] "Exfoliation corrosion resistance of near peak-aged AA7449 as a function of quench speed and aging treatment" T.J. Warner, R. Dif, P. Lequeu, P. Lassince (1998) Proc. of 6th Intl.conference on aluminium alloys: their physical and mechanical properties, Toyohashi, Japan, July 5-10 1998, pp 1669-1674

2. Materials and methods

2.1 Materials

2.1.1 Plate references

The references and composition of the plates characterized in this study are presented in table 2.1.

Table 2.1. Plates considered in this study

Plate no.	Th (mm)	Cast no.	Ingot	Si	Fe	Cu	Mg	Zn	Zr	Ti
807450	39.5	92856	66			1.97	2.165	8.375	0.1134	
708695	23	99379	25	0.045	0.068	1.95	2.27	8.31	0.105	
716937	30	90156	66	0.049	0.075	1.87	2.35	8.38	0.114	0.03

2.2.2 Aging treatments

The initial mechanical characterizations were performed on plate 807450 (in TAF temper) in the following two tempers:
T6x51: 6h @ 110°C + (ramp 16°C/h) 24h @ 135°C
T7951: (ramp 16°C/h) 17h @ 150°C

On the other two plates (716937 et 708695), a larger number of aging conditions were tested in an attempt to identify a condition combining good corrosion resistance with the required CYS. These are listed in table 2.2.

Table 2.2. Detailed aging conditions for the second testing campaign. Note that the third aging step of 24h @ 120°C was performed as a separate treatment (i.e. after cooling to ambient temperature). The ramp between 1st and 2nd aging steps was performed at 16°C/h

Repère des échantillons	Effets étudiés	tps (h)	T (°C)	ts (h)	T (°C)	tps (h)	T (°C)
A	Température du 1er palier	24	80	24	135		
B		24	100	24	135		
C		24	120	24	135		
D	temps et température du 2ème palier	24	100	18	140		
E		24	100	7	150		
F		24	100	2.5	160		
G		24	100	30	140		
H	3ème palier	24	100	18	140	24	120
I		24	100	7	150	24	120
J		24	100	2.5	160	24	120

2.2 Test methods

2.2.1 Tensile and compressive curves

All the static testing specimens were sampled at mid-thickness of the plates. The following samples were used:
For compression testing, cylindrical specimens 12 mm in diameter, length 23 mm;
For tensile testing, 10 mm diameter specimens (reference TOR10 at CRV).

2.2.2 R-curve testing

These tests were performed according to [redacted]'s requirements, i.e. on L-T CCT specimens sampled at T/2, W = 406 mm and B = 6.3 mm, $2a_o$ = 97.5 mm (see specimen drawing in the appendix).

2.2.3 Corrosion tests

Two types of corrosion test were performed: EXCO (according to ASTM G34) and DCB (see specimens in appendix). Detailed experimental conditions and results are provided in corrosion file number 4017.

3. Results

3.1 Preliminary characterization of plate 807450

This plate was characterized in two tempers (T7951 and T6x51 as defined above) in tension, compression, EXCO, and DCB. Moreover, two R-curves were obtained on the T7951 temper. A summary of the tension and compression results is provided in tables 3.1 and 3.2 below. The R-curve results are summarized in table 3.3. The corrosion data are presented separately in §3.3.

These data demonstrate that the levels of CYS(L) obtained for the T6x51 temper are roughly in accordance with [redacted] requirements. However, the corrosion resistance of this temper is clearly insufficient (the T6x51 temper ranked at EC, T7951 at EB). In an attempt to obtain a more satisfactory corrosion performance, a second series of aging treatments and basic characterizations (CYS(L) and ECXO) was performed (see results in §2.3).

Table 3.1. Summary of tensile data on plate 807450

Refs	Temper	807450 39.5 mm							
		TYS MPa		UTS MPa		El% F max		El% rupt	
		m	SD	m	SD	m	SD	m	SD
50-ML6	T6x51	623	1	652	2	6.84	0.36	11.18	0.42
50-ML7	T7951	598	1	629	1	6.31	0.13	11.27	0.46

(mean and standard deviation of three datapoints)

Table 3.2. Summary of compressive data on plate 807450

Refs	Temper	807450 39.5 mm			
		CYS MPa		Young's modulus MP	
		m	SD	m	SD
50-ML6	T6x51	611	3	70783	2234
50-ML7	T7951	589	2	72016	1911

(mean and standard deviation of three datapoints)

Table 3.3. Summary of CCT toughness data (7449-T79)

Specimen reference	Kc effective (MPa$\sqrt{m}$)	K apparent (MPa$\sqrt{m}$)
50 L-T1	91.0	84.5
50 L-T2	111.2	87.4

3.2 Detailed study of aging treatments

Three approaches to improving the EXCO resistance of 7449-T6x51 were evaluated: optimizing the first aging step in order to minimize grain boundary decoration during aging (treatments A, B, C, with first steps of 24h at respectively 80°C, 100°C and 120°C); optimizing the temperature of the second step at identical equivalent aging time (see treatments D, E, F, and G (slightly longer equivalent time); and the use of a third aging step at 120°C (derived from T77 approach, but distinct as the second step is performed at a lower temperature, see U, I, and J below).

The results obtained on these tempers are presented in detail in appendix 3. The CYS(L) data are also illustrated graphically in figure 3.1 below. The following points should be noted:

- none of the aging treatments gives an improvement in EXCO performance compared to the "baseline" T6x51 ;
- there are no clear trends of CYS(L) with the temperature of the first aging step, nor with the temperature of the second aging step ;
- the performance of a third step increases significantly CYS(L) for both plates ;
- the performance of plate 708695 after a second step of 30h @ 140°C is apparently inconsistent both with the same plate's T7951 performance and with the CYS(L) of 716937 after the same aging treatment.

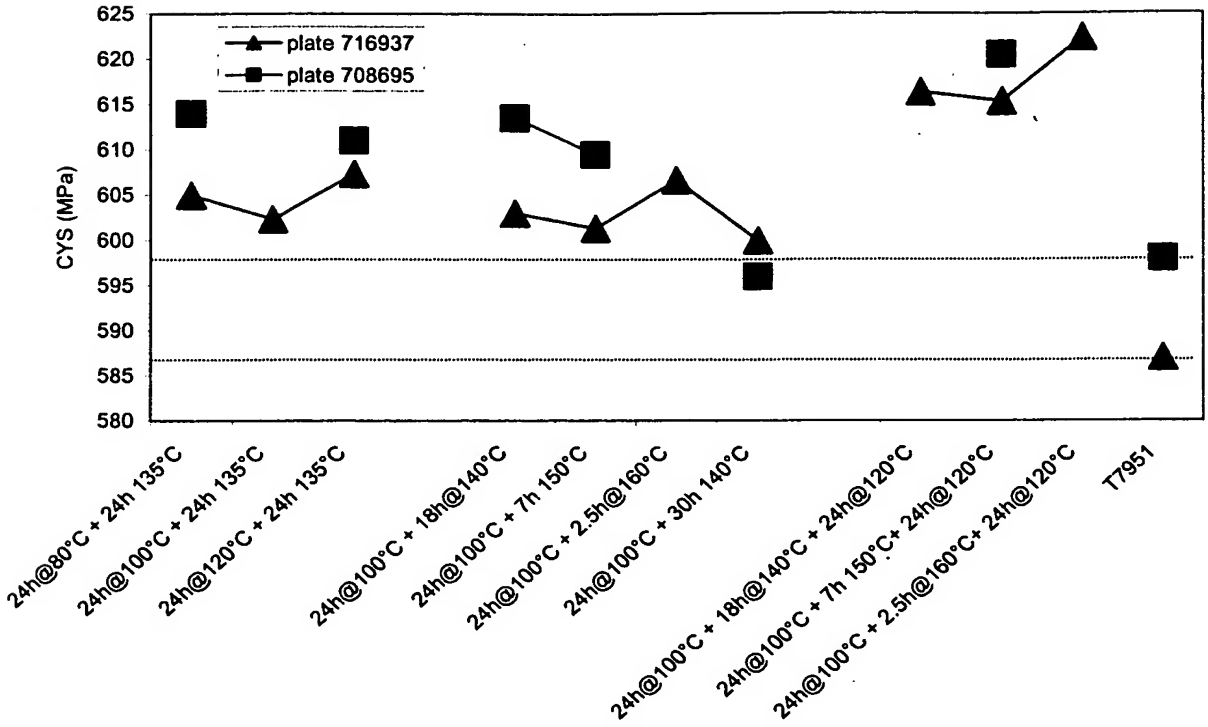


Figure 3.1. Illustration of CYS(L) as a function of aging treatment. See text for discussion.

3.3 DCB (Double Cantilever Beam) results

The results of the DCB experiments are summarized in figure 3.2, and compared with previously obtained DCB data in figure 3.3. The T6x51 temper (in this case second step of 24h@135°C) performs less well than the T7951 temper in these tests. The results obtained in the present study do not appear to be consistent with those obtained in previous testing campaigns. It should be noted that the previous 7449 data (see lot 1 in figure 3.3) were obtained on a 25 mm plate rolled from a 300 mm thick ingot (#25).

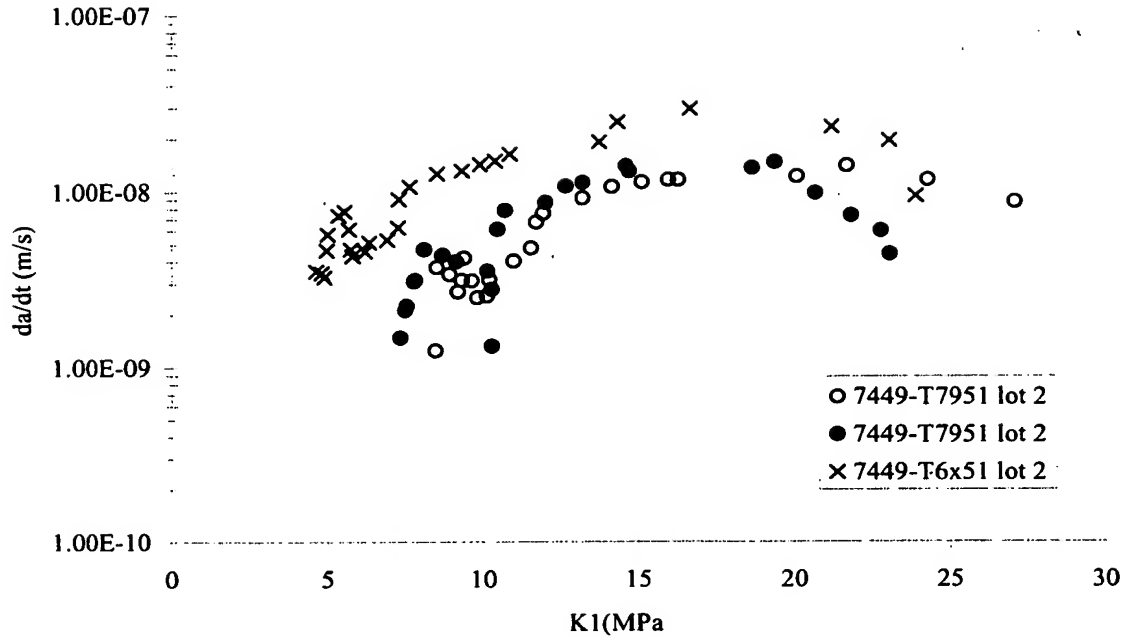


Figure 3.2. Graphical representation of DCB data acquired in the course of the present study (lot 2 = plate no 807450). See corrosion file 4017 for detailed results and testing conditions. See also schematic diagram in annex 3.

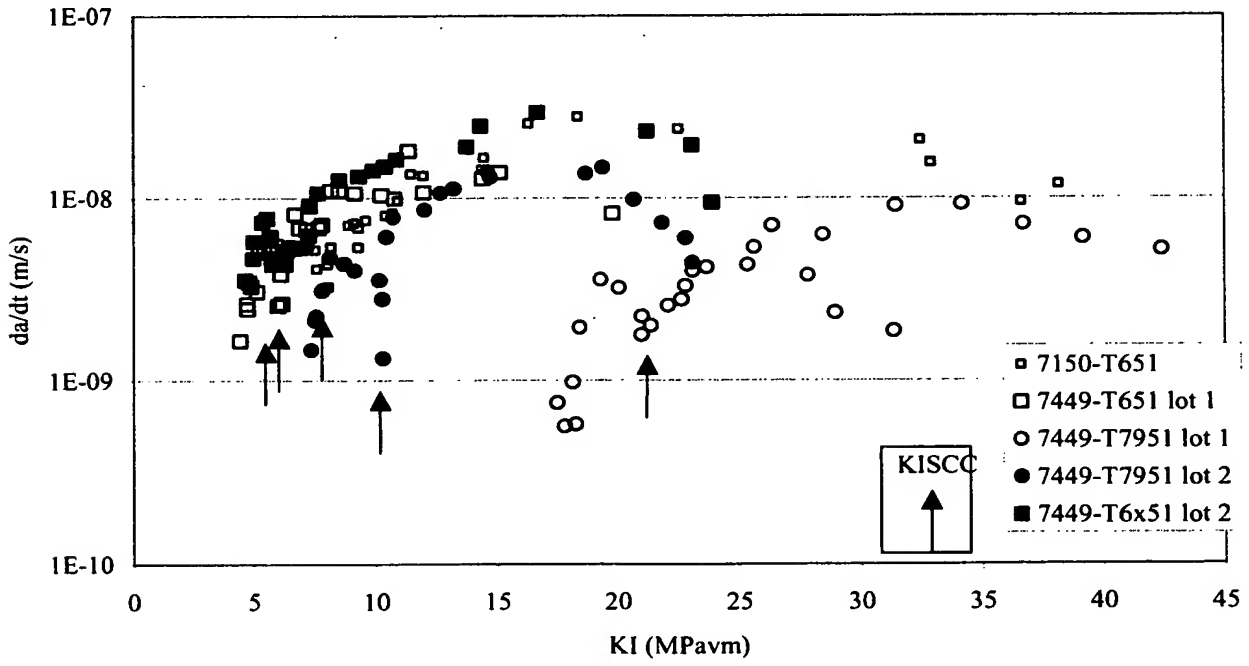


Figure 3.3. Comparison of recent DCB data with previous results on wing skin plate (7449-T651, 7449-T7951, and 7150-T651). The positions of the K_{ISCC}(S-L) values for each product are indicated on the figure with an arrow. The acceleration of the crack growth subsequent to the K_{ISCC} estimations (i.e. at lower K_I) is associated with crack wedging phenomena due to the corrosion product. See also schematic diagram in annex 3.

4. Discussion

4.1 Evaluation of properties vs goals

A comparison of the properties achieved with the various tempers of 7449 with [redacted]'s property goals (see table 4.1) indicates that neither of the solutions currently available would meet [redacted]'s requirements. The T6x51-type tempers are very close to the CYS(L) target but do not meet the EXCO target, nor probably the toughness target[3]. The T7951 temper would meet the EXCO target but is otherwise insufficient in CYS(L) and toughness.

Table 4.1. Comparison of properties of T6x51, T7951 tempers with [redacted] goals (typicals)

Property	Property goal (SI)	7449-T6x51	7449-T7951 properties
CYS(L) T/2 sampling	613 MPa	610 MPa	595 MPa
K_{app}(L-T) T/2, B = 6.35 mm, W = 406 mm	101 MPa$\sqrt{m}$	?	86 MPa$\sqrt{m}$
Exfoliation corrosion (ASTM G34)	EB	EC	EB

The other interesting point in the current work was the performance of the three-step aging treatments, which are significantly better in CYS(L) though not in EXCO for a given equivalent aging time (see figure 4.1). For a given equivalent time, the third step enables an increase in CYS(L) of approximately 15 MPa. We did not observe so marked an effect on tensile yield strength with similar treatments on 7449 previously (see CRV/ALT/TJW 97.200), although in the previous study generally higher second step temperatures (T7951 + 24h @ 120°C, and three step treatments with second steps at 165°C to 176°C) and greater equivalent aging times were used.

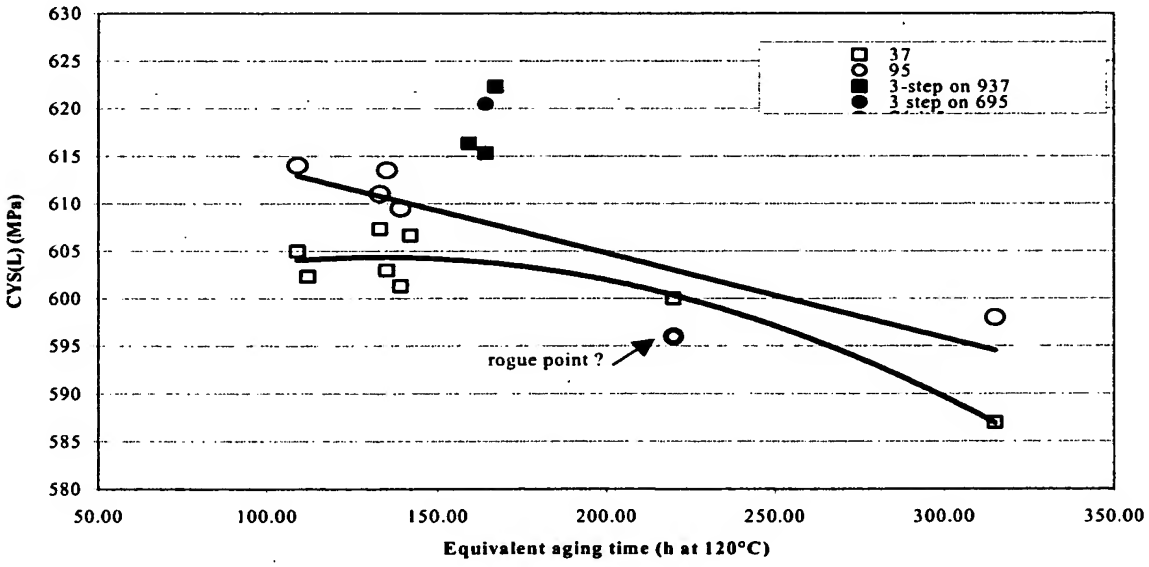


Figure 4.1. CYS(L) for the aging treatments of §3.2 as a function of equivalent aging time[4] at 120°C .

[3] This point would merit verification : our previous results on 7449 (see CRV/ALT/TJW 98.055), consistent with the literature (e.g. Kirman 1975), suggest that for a given strength lower temperature aging treatments give better toughnesses.

[4] Equivalent aging time is approximated by the following equation : $t_{eq} = \frac{\int_{Tcycle} \exp(\frac{-Q}{RT})dt}{\exp(\frac{-Q}{RT_{ref}})}$ where Q/R = 15866 K, and T is in kelvin).

4.2 Directions for property improvements

(REDACTED)

5. Conclusions

The current work demonstrates that 7449-T6x51 would meet [redacted]s CYS(L) requirement. However, it would not achieve the EXCO target, and probably not the toughness target (see table 4.1). 7449-T7951 plate meets the EXCO target but is currently insufficient in both CYS(L) (by about 18 MPa) and K_{app}(L-T) (by about 15MPa$\sqrt{m}$).

Further conclusions of this study include the following:

- for the T6x51-type tempers there are no clear variations of CYS(L) with the temperature of the first aging step, nor with the temperature of the second aging step for a given equivalent aging time;
- the performance of a third aging step increases significantly CYS(L) for both plates;
- none of the modifications in the T6x51 aging treatment showed an improvement in EXCO performance;
- the SCC performance of 7449-T6x51 is inferior to that of 7449-T7951 as expected. However, there remains an unexplained inconsistency between these recent results and those obtained previously.

Some directions for property improvements are proposed, including:

(REDACTED)

Annexe 1

Chemical composition of the plate- alloy 7449

Transformation sequence and heat treatments

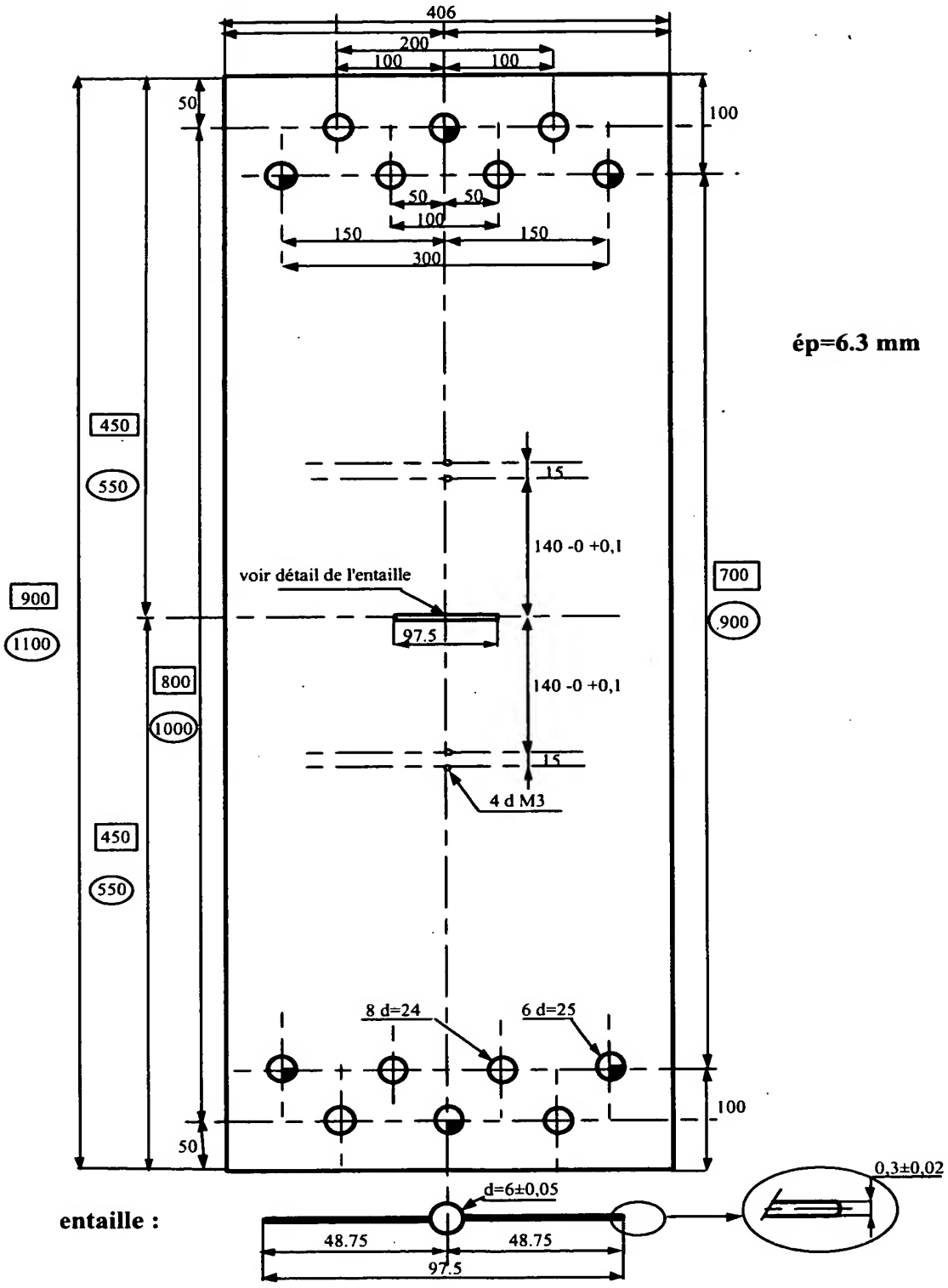
No. Lot	Plate	ROLLING DIMENSIONS		HOMOGENIZATION			REHEATING					ROLLING		
		l(mm)	thickness (mm)	furnace	setpoint	actual		setpoint		actual		temperature		
							furnace	increase	holding	increase	holding	setpoint	start	end
173375/042	807450	3180	40.8	127	48+5-0 h at 473+0-3°C	48 h at 470-472°C	216	17h at 475°C	3h at 450°C	17h at 475°C	6h15 at 450°C		432	410

No. Lot	Plate	SOLUTION HEAT TREATMENT			Waiting time between quenching and stretching	STRETCHING		ARTIFICIAL AGING		
								First step		
		Quench ref.	setpoint	actual		setpoint	actual	heat treatment lot	setpoint	actual
173375/042	807450	4169	12h at 174+1-2°C	12h01 at 472-475.9°C	1h45		2.8-2.6-2.3	0516	22h at 118-124°C	22h at 118-124°C

No. Lot	Plate	MECHANICAL PROPERTIES Controlled Stretching													CONDUCTIVITY					
									compression		EXCO		periodic testing		internal stress	Temper TAF				Temper T7951	
		direction	Rm MPa		Rp 0.2 MPa		A%		K1C MPam 0.5	direction	Rp 0.2 MPa	S	H86	direction	CST	Wtotal kJ/m3	Min	Max	Av. FS	Av. F1	Res. Lab	Metal. Lab
173375/042	807450	LL1 TAF (1/4 cp)	630	630	593	593	11.24	11.24									17.8	18.8	18.4	18.2		
		LL4 TAF (½ cp)	668	668	625	625	11.88	11.88														
		LL2 (1/4 cp)	616	617	594	596	11.31	10.9	23.87													
		LL3 (1/4 cp)	618	1.4	598	2.8	10.49	0.6														
		LL5 (½ cp)	638	638	613	612	10.96	11														
		LL6 (½ cp)	637	0.7	611	1.4	10.88	0.1														
		TL1 (1/4 cp)	626	627	594	594	9.17	9	22.62													
		TL2 (1/4 cp)	627	0.7	594	0.0	9.02	0.1														
		TL3 (½ cp)	617	618	590	592	9.31	10														
		TL4 (½ cp)	619	1.4	593	2.1	10.64	0.9														

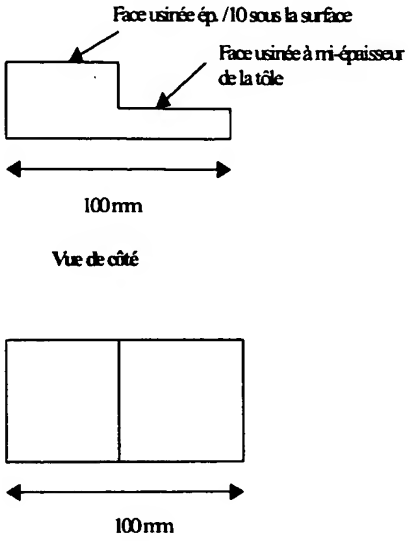
Eprouvette CCT W = 406 mm
suivant AIMS 03-02-018

figure 4

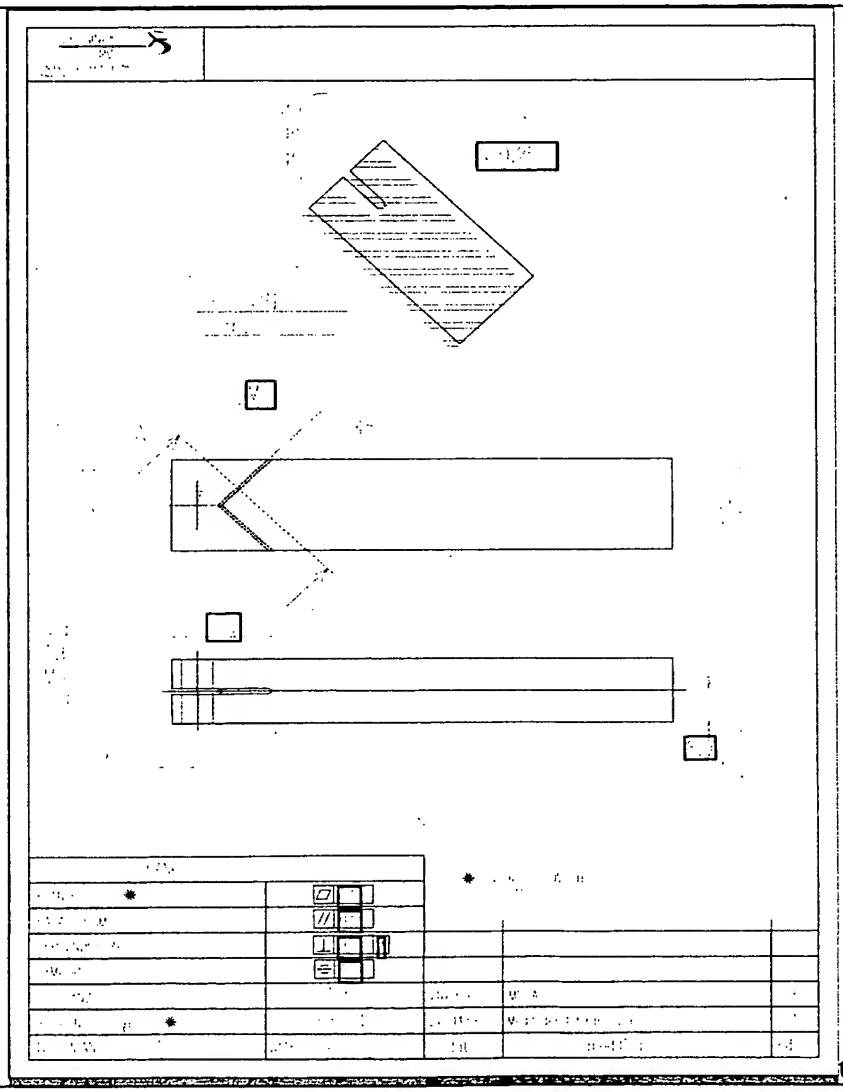


ép=6.3 mm

entaille :



Test EXCO



test DCB

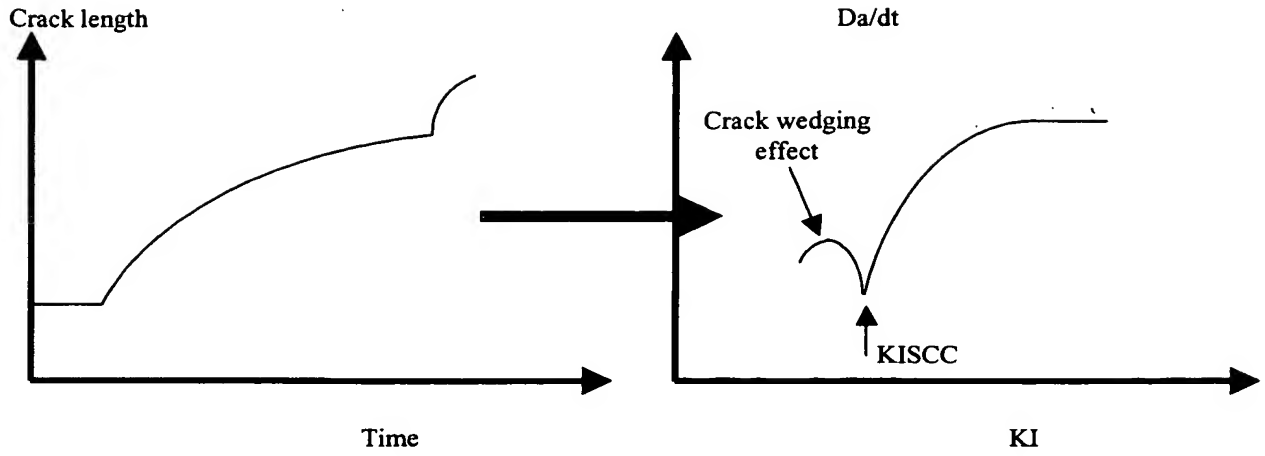


Figure A1 – schematic representation of the results of a DCB experiment (left) and the da/dt vs stress intensity factor curve that is constructed from such results. See also results in text.

Annexe 3

Aging treatment						COMPRESSION RESULTS							EXCO
1st step		2nd step		3rd step		Rep	R0.2 (L)			Module (L)			result
t(h)	T(°C)	t(h)	T(°C)	t(h)	T(°C)			m	SD		m	SD	Ranking
24	80	24	135			37A L1	614	**605**	**8**	71471	**70281**	**1788**	EC*
						37A L2	600			68225			
						37A L3	601			71148			
24	100	24	135			37B L1	606	**602**	**4**	70447	**71200**	**1387**	EC*
						37B L2	599			70353			
						37B L3	602			72801			
24	120	24	135			37C L1	619	**607**	**10**	74603	**72335**	**2013**	EC*
						37C L2	602			70759			
						37C L3	601			71642			
24	100	18	140			37D L1	607	**603**	**5**	71511	**70598**	**876**	EC*
						37D L2	604			70518			
						37D L3	598			69765			
24	100	7	150			37E L1	601	**601**	**4**	72142	**70618**	**1320**	EC*
						37E L2	598			69851			
						37E L3	605			69862			
24	100	2.5	160			37F L1	606	**607**	**2**	71376	**72302**	**1745**	EC*
						37F L2	605			71216			
						37F L3	609			74315			
24	100	30	140			37G L1	602	**600**	**2**	73363	**72806**	**768**	EC*
						37G L2	600			73125			
						37G L3	598			71930			
24	100	18	140	24	120	37H L1	616	**616**	**3**	70805	**71621**	**960**	EC*
						37H L2	614			72679			
						37H L3	619			71378			
24	100	7	150	24	120	37I L1	619	**615**	**4**	70219	**70862**	**651**	EC*
						37I L2	612			71521			
						37I L3	615			70847			
24	100	2.5	160	24	120	37J L1	625	**622**	**6**	73353	**72569**	**1731**	EC*
						37J L2	626			73769			
						37J L3	616			70585			
24	80	24	135			95A L1	614	635	30	73029	**72910**	**168**	EC*
						95A L2	656			72791			
24	120	24	135			95C L1	611	**611**	**0**	72219	**72222**	**4**	EC*
						95C L2	611			72224			
24	100	18	140			95D L1	613	**614**	**1**	73592	**73244**	**493**	EC*
						95D L2	614			72895			
24	100	7	150			95E L1	606	**610**	**5**	71173	**72349**	**1663**	EC*
						95E L2	613			73525			
24	100	30	140			95G L1	592	**596**	**6**	70945	**70181**	**1080**	EC*
						95G L2	600			69417			
24	100	7	150	24	120	95I L1	619	**621**	**2**	72151	**71303**	**1199**	EC*
						95I L2	622			70455			

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